

**Standard Operating Procedure and Checklist of Minimal Requisite Facilities for utilization
of hazardous waste under Rule 9 of the Hazardous and Other Wastes (Management and
Transboundary movement) Rules, 2016**

**Utilization of Spent TiO_2 -NaCl cake generated from process residue/waste containing
chloride from the catalyst manufacturing industries for recovery of Titanium Dioxide**



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Utilization of Spent TiO₂-NaCl cake generated from process residue/waste containing chloride from the catalyst manufacturing industries for recovery of Titanium Dioxide

Procedure for grant of authorization by SPCBs/PCCs for utilization of Hazardous waste

- 1) While granting authorisation for utilization of hazardous wastes, SPCBs/PCCs shall ensure that authorisation is given only to those wastes for which SoPs on utilisation have been circulated by CPCB ensuring the following:
 - a. The waste (intended for utilization) belongs to similar source of generation as specified in SoP.
 - b. The utilization shall be similar to as described in SoP.
 - c. End-use/product produced from the waste shall be same as specified in SoP.
 - d. Authorisation shall be granted only after verification of details and minimum requisite facilities as given in SoP.
 - e. Issuance of passbooks for maintaining records of receipt of spent acid for utilization.
- 2) After issuance of authorization, SPCB shall verify the complaisance of checklist and SoP on quarterly basis for initial 2 years; followed by random checks in the subsequent period for atleast once a year.

In-case of lack of requisite infrastructures with the SPCBs/PCCs, they may engage 3rd party institutions or laboratories having EPA/NABL/ISO17025 accreditation/ recognition for monitoring and analysis of prescribed parameters in SoPs for verification purpose.
- 3) SPCBs shall provide half yearly updated list of units permitted under Rule 9 of Hazardous & Other Wastes (Management & Trans-boundary Movement) Rules, 2016 (HOWM Rules, 2016) to CPCB and also upload the same on SPCB website, periodically. Such updated list shall be sent to CPCB on a half yearly basis i.e. by July and January respectively.
- 4) Authorisation for utilisation shall not be given to the units located in the State/UT where there is no Common TSDF, unless the unit ensures authorised captive disposal of the hazardous waste (generated during utilisation) or its complete utilisation or arrangement of sharing with any other authorised disposal facility.
- 5) In case of the utilization proposal is not similar with respect to source of generation or utilization process or end-use as outlined in this SoP, the same may be referred to CPCB for clarification/conducting trial utilization studies and developing SoPs.
- 6) The source and work zone standards suggested in the SoP are based on the E(P)A notified and OSHA standard respectively, however, SPCB/PCC may impose more stringent standards based on the location or process specific conditions.

55.0 Utilization of spent TiO₂-NaCl cake

Type of Hazardous Waste	Source of Generation	Recovery/ Product
Spent TiO ₂ -NaCl Cake	Process Residue/waste containing chloride from Catalysts Manufacturing industries	Recovery of Titanium Dioxide

Utilization of Spent $\text{TiO}_2\text{-NaCl}$ cake generated from process residue/waste containing chloride from the catalyst manufacturing industries for recovery of Titanium Dioxide

55.1 Source of Waste

The spent $\text{TiO}_2\text{-NaCl}$ Cake is generated from Process Residue/waste containing chloride from Catalysts manufacturing industries and is categorized as Hazardous waste listed at 21.1 in Schedule I of HOWM Rules, 2016 which are required to be disposed off in authorized hazardous waste disposal facility in accordance with authorization condition, when not utilized as resource recovery.

Table 1- *Typical characteristics of spent $\text{TiO}_2\text{-NaCl}$ cake*

S. No.	Properties/Parameters	Unit	Typical characteristics
1	Appearance	--	Off white coloured
2	TiO_2	%	78.5
3	Salt	%	6.30
4	Moisture	%	15.2
5	Specific Gravity	gm/cm^3	4.2
6	pH	--	7.0

55.2 Utilization Process

The $\text{TiO}_2\text{-NaCl}$ cake generated from Process Residue/waste containing chloride from Catalysts manufacturing industries. The recovery of Titanium Dioxide from spent $\text{TiO}_2\text{-NaCl}$ cake mainly includes washing, filtration, drying to remove moisture, calcination with oxidation and pulverization.

Spent $\text{TiO}_2\text{-NaCl}$ cake is fed in the HDPE tank fitted with stirrer for preparation of slurry. Spent $\text{TiO}_2\text{-NaCl}$ cake contains about 15-16% moisture content. Washing of spent $\text{TiO}_2\text{-NaCl}$ cake is followed by filtration. Material is transferred to filter press through transfer pump.

The wastewater generated from filtration process is collected in the collection tank; then pumped to evaporator. The evaporator (shell and tube type) evaporates the wastewater using thermic fluid heater. Thermic fluid is circulated in the outer and opening is provided on top of the evaporator. After few batches of operations of evaporator, the side covers of the evaporator shall be opened and the residues from the evaporator are scrapped out from time to time, collected and stored in designated place before disposal to CHWTSDF.

After, filtration, the wet cake is allowed for drying. Provision of Centrifuge (optional) for removal of moisture from the washed cake is in place of/or in addition of natural drying of washed cakes. Provision of monsoon covers for the drying platform shall be provided in case of natural drying of washed cake.

The dry cake is calcined in the rotary dryer, which shall be provided with two suction hoods at the mouth of the rotary dryer for the collection of fumes escaping from the dryer during the calcination process and for the collection of dust arising during charging and unloading of rotary dryer. Both the hoods shall be provide with adequate air pollution control device (APCD) and proper stack with ID fan as prescribed by SPCB/PCC.

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Dry cake is heated up to 850°C . Pure oxygen is purged for 30 to 45 minutes duration for whitening purpose. Calcined mass is discharged after the desired temperature by tilting SS batch types rotary and recharge the same for next batch.

Calcined mass is transferred to SS trolley and spread on natural cooling platform for cooling the same up to room temperature.

Cooled lumps are pulverized in pulveriser and 250 to 300 mesh powder recovered as the final product (Titanium Dioxide) which is packed in a plastic inner liner bags.

Typical flow diagram for utilization of spent $\text{TiO}_2\text{-NaCl}$ cake for recovery of Titanium Dioxide is shown in Figure below:

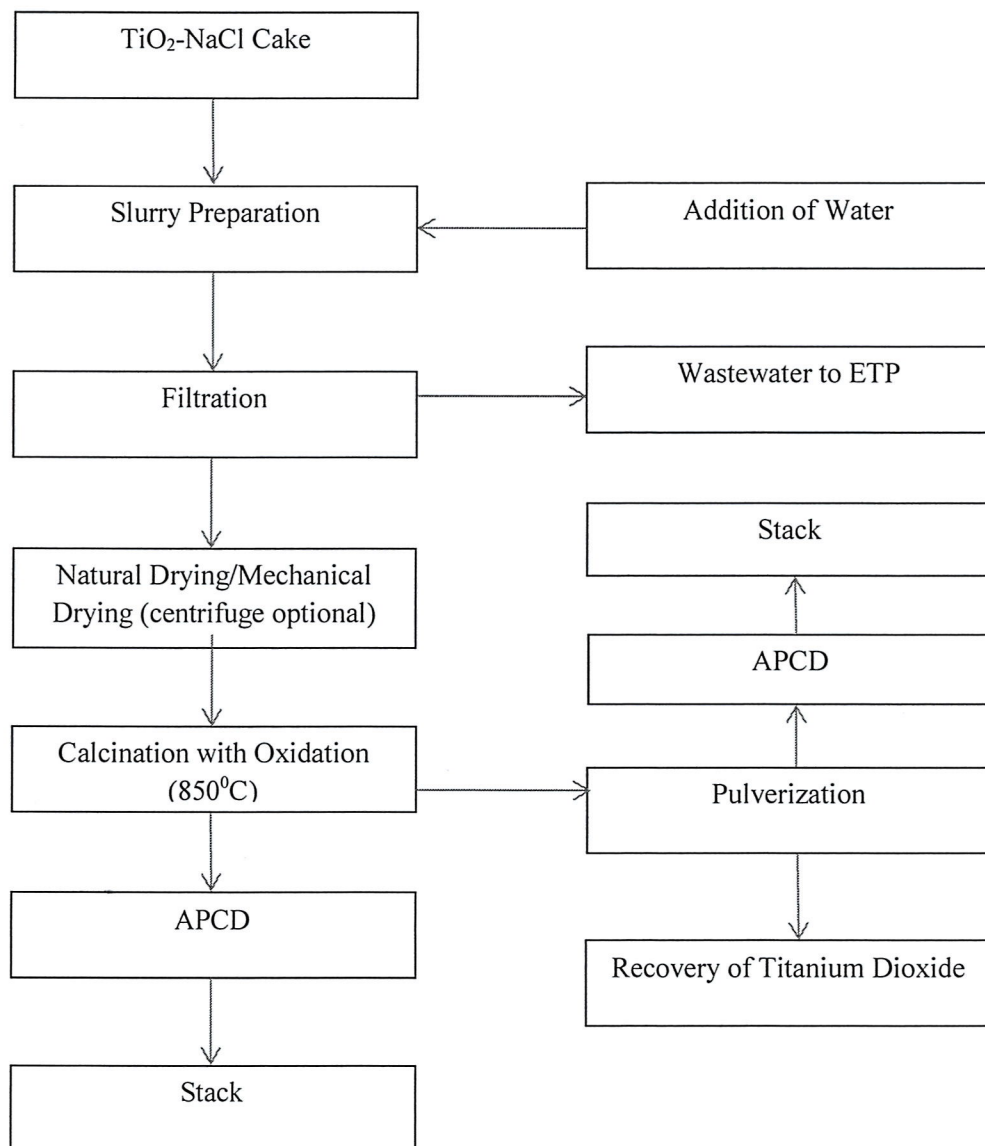


Figure: Process flow diagram for utilization of $\text{TiO}_2\text{-NaCl}$ Cake

Utilization of Spent $\text{TiO}_2\text{-NaCl}$ cake generated from process residue/waste containing chloride from the catalyst manufacturing industries for recovery of Titanium Dioxide

55.3 Product Usage / Utilization

The product i.e. Titanium Dioxide shall comply as per Bureau of Indian Standards of further respective utilization.

The unit shall label its product (i.e. Titanium Dioxide) manufactured by utilizing aforesaid hazardous waste as “This Titanium Dioxide has been manufactured by utilizing spent $\text{TiO}_2\text{-NaCl}$ cake generated from Process Residue/waste containing chloride from Catalysts Manufacturing industries”.

55.4 Standard Operating Procedure for Utilization

This SoP is applicable only for utilization of spent $\text{TiO}_2\text{-NaCl}$ cake generated from Process Residue/waste containing chloride from Catalysts Manufacturing industries, for recovery of Titanium Oxide.

1. The unit shall provide separate storage area at designated place with proper cover for storage of spent $\text{TiO}_2\text{-NaCl}$ cake.
2. The entire process area shall have dedicated leak-proof floor tiles with adequate slope to collect spillage, if any, into a collection pit. The spillages from collection pit shall be transferred to ETP.
3. It shall be ensured that spent $\text{TiO}_2\text{-NaCl}$ is procured from the industries that have valid authorization for the same from the concerned SPCBs/PCCs as required under Hazardous and Other Wastes (Management & Trans-boundary Movement) Rules, 2016. $\text{TiO}_2\text{-NaCl}$ cake shall be transported in SPCB/PCC authorized container mounted on vehicles fitted with requisite safeguards ensuring no spillage.
4. The $\text{TiO}_2\text{-NaCl}$ shall be packed in plastic bags with inner liner.
5. Transportation of spent $\text{TiO}_2\text{-NaCl}$ cake shall be carried out by sender or receiver (utilizer) only after obtaining authorization from the concerned SPCBs/PCCs under the Hazardous and Other Wastes (Management & Trans-boundary Movement) Rules, 2016.
6. The unit shall provide fixed pipeline connection for transferring the wastewater.
7. Treatment and disposal of wastewater:

Following are the sources of wastewater from utilization process;

- a. Wastewater (generated from floor washing/reactor wash/vehicle wash/spillages, etc.)
- b. Washing of spent $\text{TiO}_2\text{-NaCl}$ cake
- c. Filtrate from filter press

Source of wastewater shall be inter connected with ETP for Physico-Chemical treatment so as to comply with the prescribed standards, in case of CETP or be treated in captive ETP having adequate treatment facilities to comply with surface water discharge standards as stipulated in the Consent issued by the SPCBs/PCCs.

In case of zero discharge condition by SPCB/PCC, the treated waste water from ETP may be managed as per conditions stipulated by the SPCBs/PCCs.



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8. The treated effluent shall be discharged in accordance with the conditions stipulated in the Consent to Operate issued by concerned SPCB/PCC under the Water (Prevention and Control of Pollution) Act, 1974.
9. There shall be provision of centrifuge (optional) for efficient removal of moisture from the washed cake in place of/or in addition to natural drying of washed cake.
10. The unit shall provide proper suction system for the hoods provided for the rotary dryer with proper stack and adequate stack monitoring facility.
11. The unit has to provide adequate Air Pollution Control Device (APCD) (i.e. like proper bag filter with casing) for the pulverizer and rotary dryer and proper stack with adequate stack monitoring facility.
12. The stack attached to the dust extraction system shall be dispersed into atmosphere through minimum stack height of 6 m above the roof top or as prescribed by the concerned SPCB/PCC, whichever is more.
13. The unit shall maintain proper ventilation in the work zone and process areas. All personnel involved in the plant operation shall wear proper personal protective equipment (PPE) specific to the process operations involved and type of chemicals handled as per Material Safety Data Sheet (MSDS). The safety precautions of the worker shall be in accordance with the Factory Act, 1948, as amended from time to time.
14. SPCBs/PCCs shall ensure synchronization of generation and utilization of spent TiO₂-NaCl cake and the same shall be reflected in respective authorization specifying name and quantity.
15. Prior to utilization of spent TiO₂-NaCl cake; the unit shall obtain authorization for generation, storage and utilization of spent TiO₂-NaCl cake from the concerned SPCB/PCC under the Hazardous and Other Wastes (Management and Trans-boundary Movement) Rules, 2016.
16. The unit shall submit quarterly and annual information on hazardous wastes consumed, its sources, products generated or resources conserved (specifying the details like type and quantity of resources conserved) to concerned SPCB/PCC.
17. The hazardous wastes generated (namely the filter residue, ETP sludge, Evaporation residue generated from dryer/forced evaporator, product spillages, damaged filter liners, etc.) shall be collected and temporarily stored in non-reactive drums/bags under a dedicated hazardous waste storage area and be sent to authorized common TSDF or other authorized facility within 90 days from generation of the waste in accordance with the authorization issued by the concerned SPCB/PCC. Such storage area shall be covered with proper ventilation.
18. In case of environmental damages arising due to improper handling of hazardous wastes including accidental spillage during generation, storage, processing, transportation and disposal, the unit shall be liable to implement immediate response measures, environmental site assessment and remediation of contaminated soil/groundwater/sediment etc. as per the "Guidelines on Implementing Liabilities for Environmental Damages due to Handling & Disposal of Hazardous Wastes and Penalty" published by CPCB.



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19. The unit shall ensure that the said utilization process and its associated activities shall be demarcated separately within the unit.
20. The unit shall provide suitable fire safety arrangements and flame proof electrical fittings.
21. The unit shall provide personal protective equipment like aprons, safety hand gloves, and safety shoes, helmets to the persons involved in production activities and in handling hazardous wastes.
22. During the process of utilization and handling of hazardous waste, the unit shall comply with the requirements in accordance with the Public Liability Insurance Act, 1991 as amended, wherever applicable.

55.5 Record/Returns Filing

1. The unit shall maintain a passbook issued by concern SPCB, wherein the following details of each procurement of spent TiO₂-NaCl Cake shall be entered:
 - Address of the sender
 - Date of dispatch
 - Quantity procured
 - Seal and signature of the sender
 - Date of Receipt in the premises
2. A log book with information on source and date of procurement of spent TiO₂-NaCl Cake, quantity, date wise utilization of the same, hazardous waste generation and its disposal, etc. shall be maintained including analysis report of fugitive emission monitoring & effluent discharged, as applicable.
3. The unit shall maintain record of hazardous waste utilized, hazardous waste generated and disposed as per Form 3 & shall file annual returns in Form 4 as per Rule 20 (1) and (2) of the Hazardous and Other Wastes (Management and Transboundary Movement) Rules, 2016, to concerned SPCB/PCC.
4. The unit shall submit quarterly and annual information on hazardous wastes consumed, its source, products generated or resources conserved (specifying the details like, type and quantity of resources conserved) to the concerned SPCB.

55.6 Standards

1. Source Emissions from the stack attached to dust extraction system of rotary dryer (Calcination process) and pulverizer shall comply with the following standards or as prescribed by the concerned SPCB/PCC, whichever is stringent;

Parameters	Unit	Prescribed Limit
Particulate Matter	mg/Nm ³	50
NO _x	ppm	50
SO _x	ppm	100
CO	mg/Nm ³	100



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2. Fugitive emission in the rotary dryer and pulverizer area shall comply with the following standards

PM_{10}	5 mg/m^3 TWA* (PEL)
TiO_2 dust	15 mg/m^3 TWA* (PEL)

PEL: Permissible Exposure limit

*time-weighted average (TWA): measured over a period of 8 hours of operation of process.

(Reference: Occupational Safety and Health Standards 1910: 1000);

3. The monitoring shall be carried out by NABL or ISO 17025 accredited/EPA approved laboratories and the results shall be submitted to the concerned SPCB/PCC on a quarterly basis.
4. Standard for wastewater discharge: Treated effluent shall be discharged in accordance with the conditions stipulated in Consent to Operate issued by respective SPCB/PCC under the Water (Prevention and Control of Pollution) Act, 1974.

55.7 Siting of Industry

Facilities for utilization of spent TiO_2 -NaCl cake shall be located in a notified industrial area or industrial park/estate/cluster and in accordance with Consent to Establish issued by the concerned SPCB/PCC.

55.8 Size of Plant and Efficiency of Utilization

1. About 0.794 MT of Dry TiO_2 would be recovered from 1 MT of spent TiO_2 -NaCl cake in batch process.
2. About 0.845 MT of Dry TiO_2 would be recovered from 1 MT of yield of filtered cake from the filter press.
3. About usage of 170 litres of water in washing of 1 MT of TiO_2 -NaCl cake and about 325 litres of wastewater would be generated (filtrate) per 1 MT of TiO_2 -NaCl cake.

Therefore, requisite facilities of adequate size of storage shed and other plant & machineries as given in 55.10 below shall be installed accordingly.

55.9 On-line detectors / Alarms / Analysers

Online emission monitoring system shall be installed with the data transmission to CPCB and SPCBs server in case of continuous process operations for PM.

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55.10 Check List of Minimal Requisite Facilities

Sl. No.	Particulars
1.	Covered Hazardous Waste storage space for storage of spent TiO ₂ -NaCl cake.
2.	Scrubber (With Vessel of Slurry Preparation)
3.	Filter press
4.	Centrifuges (optional)
5.	Rotary Dryer/Calcination Kiln (Dryer of adequate size operated electrically or by fuel as permitted by the concerned SPCB/PCC.
6.	Pulverizer with adequate APCD and stack as permitted by the concerned SPCB/PCC.
7.	Adequate Effluent treatment plant with fixed pipeline connection followed by Evaporator in case of in-house ETP treatment to maintain zero discharge. In case of CETP discharge conditions as prescribed by the concerned SPCB/PCC shall met.
8.	Stacks to have sampling port, platform, access to the platform etc. as per the Guidelines on Methodologies for Source Emission Monitoring published by CPCB under Laboratory Analysis Techniques LATS/20/2013-14.
9.	Dedicated separate covered hazardous waste (Filter press etc.) storage area to store hazardous waste generated during utilization process.

